

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

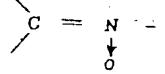
### Process for Producing Isonitrones

We, FARBFENFABRIKEN BAYER AKTIEN-GESELLSCHAFT, a body corporate organised under the laws of Germany of Leverkusen-Bayerwerk, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement: —

The present invention relates to a new class of organic compounds which contain the group



they are thus isomeric with the known nitrones or oxime-N-ethers which contain the group



For that reason the new compounds are called *isonitrones*. They differ, however, from the corresponding nitrones in their physical and chemical properties. The differences in the physical properties of the two groups of com-

pounds consist in that the melting point and the boiling point of the *isonitrones* are generally lower, in that the characteristic group of the *isonitrones* in contrast to the characteristic group of the nitrones does not yield an exaltation of the molecular refractivity and in that it does not absorb in the ultra-violet region.

The *isonitrones* differ in their chemical behaviour from the nitrones especially in that they contain active oxygen. They liberate iodine, therefore, instantly and quantitatively in proportion to their content of active oxygen from a solution of acidified potassium iodide.

Like the nitrones the *isonitrones* may be converted to the isomeric acid amides. Whereas, however, in the case of the nitrones the catalytic action of catalysts of the Beckmann conversion is necessary, the *isonitrones* may in general be converted quantitatively by themselves into acid amides by heating to somewhat elevated temperatures.

The following table gives a summary of typical *isonitrones* together with their melting points or boiling points, in degrees Centigrade.

- 50 Methyl-N-isobutyl-*isonitron*e
- n-propyl-N-methyl-*isonitron*e
- n-propyl-N-isopropyl-*isonitron*e
- n-propyl-N-cyclohexyl-*isonitron*e
- dimethyl-N-isopropyl-*isonitron*e
- dimethyl-N-cyclohexyl-*isonitron*e
- tetramethylene-N-cyclohexyl-*isonitron*e
- pentamethylcyclohexyl-*isonitron*e
- pentamethylene-N-isobutyl-*isonitron*e
- pentamethylene-N-cyclohexyl-*isonitron*e
- di-pentamethylene-N,N'-ethylene-di-*isonitron*e
- pentamethylene-N-phenyl-*isonitron*e
- (3-methyl-pentamethylene)-N-phenyl *isonitron*e
- pentamethylene-N-(p-chlorophenyl)-*isonitron*e
- phenyl-N-methyl-*isonitron*e
- methyl-phenyl-N-cyclohexyl-*isonitron*e
- phenyl-N-isopropyl-*isonitron*e
- furyl-N-methyl-*isonitron*e

	Boiling point	Melting point
	40—42°/32mm	
	42—44°/32mm	
	43—45°/13mm	
	69°/0.01mm	
	58—59°/60mm	
	72°/8mm	
	74—76°/0.4mm	
	72—74°/8mm	
	55—57°/0.6mm	
	90—92°/0.3mm	
		106—107°
		75°
		65—66°
		69—70°
	70°/2.5mm	
	118—120°/0.8mm	
	65—67°/0.9mm	
	44—46°/0.6mm	

[Price]

Price

The *isonitriles* may be produced for instance in the following ways:

5 Aldimines or ketimines obtained in a known manner by the condensation of aldehydes or ketones with ammonia or with primary amines, i.e. products which are also known as Schiff's bases or azomethines, are reacted with per-compounds, e.g. hydrogen peroxide, or more especially with per-acids. The *isonitriles* are thus formed, probably in an analogous manner 10 to Prileschajeff's reaction, by one oxygen atom entering into one molecule of the imine.

15 Although the condensation of aldehydes and especially of ketones with primary amines leads only to equilibrium mixtures, formation of the *isonitriles* does in many cases take place without difficulty in good yield when instead of pure aldimines or ketimines a mixture of an aldehyde or ketone and a primary amine is reacted 20 with a per-compound.

25 When a per-acid is used as per-compound there may also be added to the preparation alkaline substances such as soda or potash in order that the carboxylic acids which are formed in the reaction may be neutralized. This procedure is therefore advisable in all cases in which it is intended to produce acid-sensitive *isonitriles*, for instance, *isonitriles* which are derived from aromatic amines.

30 In place of free ammonia and free primary amines the salts thereof, for instance, carbonates or acetates, may be reacted with per-compounds in the presence of aldehydes or ketones and *isonitriles* may thereby be obtained in good yield. This procedure is especially advantageous when it is intended to convert readily volatile bases such as ammonia, methylamine or ethylamine.

35 Finally, it should be mentioned that apart 40 from the process forming the object of the present invention, a more or less high proportion of *isonitriles* may be obtained according to the reaction conditions by the oxidation of primary amines with per-compounds, other reaction products being obtained in the same reaction; the mechanism of this reaction is not yet clear but possibly involves the formation of intermediate aldehydes or ketones.

45 In the new process for the preparation of *isonitriles* it is advantageous to add equimolar portions of imines, or aldehydes or ketones and primary amines, and of per-compounds.

50 It is, however, advisable in many cases to carry out the reactions in inert solvents for instance in benzene, methylene chloride, chloroform, carbon tetrachloride, ether, benzene or toluene.

55 The reaction may be accelerated by applying elevated temperatures and correspondingly increased pressures. The temperature during the conversion may vary according to conditions. Good results are obtained at temperatures ranging between about -20° C. and +50° C., the preferred range being between 60 about 0 and about +20° C. However when

the per-compound is used in the form of hydrogen peroxide it is necessary to apply higher temperatures which preferably lie between about 40° C. and 100° C.

70 When using hydrogen peroxide as per-compound it is advantageous to add catalysts such as molybdenum trioxide or tungsten trioxide or such compounds which act as stabilizers for hydrogen peroxide in alkaline solution, e.g. magnesium hydroxide or alkali metal silicates such as sodium silicate.

75 The aldimines and ketimines suitable as starting materials for the production of *isonitriles* include for instance the condensation products of aldehydes (e.g. acetaldehyde, propionaldehyde, *n*-butyraldehyde, *isobutyraldehyde*, *isovaleraldehyde*, heptaldehyde, palmitic aldehyde, succinic di-aldehyde, cyclohexyl-formaldehyde, phenylacetraldehyde, benzaldehyde, *o*-, *m*-, *p*-chlorobenzaldehyde, *o*-, *m*-, *p*-nitrobenzaldehyde, *o*-, *m*-, *p*-toluylaldehyde,  $\alpha$ , $\beta$ -naphthaldehyde and furfural) or of ketones (e.g. acetone, methyl-ethyl-ketone, methyl-propyl-ketone, diethyl-ketone, methyl-heptyl-ketone, acetophenone, *o*-, *m*-, *p*-chloro-acetophenone, cyclopentanone, cyclohexanone, 1-methyl-cyclohexanone-2, 1-cyclohexyl-cyclohexanone-2 and cyclohexanediketone-1-4), with primary amines (e.g. methylamine, ethylamine, isopropylamine, *n*-butylamine, *sec*-butylamine, *tert*-butylamine, *isobutylamine*, *isoamylamine*, dodecylamine, ethylenediamine, hexamethylenediamine,  $\epsilon$ -amino-caproic acid, cyclopentylamine, cyclohexylamine, 2-methylcyclohexylamine, 4-methylcyclohexylamine, hexahydrobenzylamine, benzylamine, aniline, *o*-, *m*-, *p*-toluidine, *p*-xylidine, *o*-, *m*-, *p*-chloroaniline).

80 Among the suitable per-compounds are mentioned hydrogen peroxide, Caro's acid, 90 organic per-acids (e.g. peroxyformic acid, peroxyacetic acid, peroxypropionic acid, peroxybutyric acid, peroxybenzoic acid and monoperoxyphthalic acid), as well as mixtures of hydrogen peroxide with organic acids (e.g. 95 hydrogen peroxide with formic acid or glacial acetic acid).

100 From such starting materials there may be obtained *isonitriles* of the aliphatic, cycloaliphatic, aromatic and heterocyclic series.

105 The *isonitriles* are capable of manifold applications and are especially suitable for the production of organic intermediates.

110 The conditions for the above mentioned conversion by rearrangement of the *isonitriles* into amides, especially the reaction temperature, depend on the constitution of the relevant *isonitriles* and may be readily determined by simple experiments. Some *isonitriles*, for instance the pentamethylene-N-phenyl *isonitrile*, change even at room temperature into the corresponding acid amides. In general the conversion may be carried out between about room temperature and about 250° C. and the corresponding pressures which are given by the 115 120 125 130

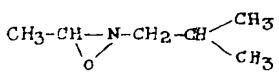
specific nature of the *isonitriones* used in any particular case. Since the quantity of heat which is liberated in the conversion is in general considerable it is advisable to use the *isonitriones* not by themselves but in the form of solutions in solvents having elevated boiling points such that the conversion readily takes place at boiling temperature. It is not necessary to use pure *isonitriones* since the crude reaction mixtures which are obtained in the manufacture of the *isonitriones* are suitable for this conversion. Solvents of the above mentioned type include for instance benzine, methylene chloride, chloroform, carbon tetrachloride, benzene, toluene, xylene, chlorobenzene, nitrobenzene, cyclohexane, tetrane, decalene, ether, dioxan and anisole.

The following Examples are given for the purpose of illustrating the invention.

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## EXAMPLE 1.

99 Grams (1 mol.) ethylidene *isobutyl* amine (boiling point 92—93° C.) in 500ml. of ether are reacted drop by drop with stirring, at 0° C. inner temperature, with 90gm. of an 85% per-acetic acid (1 mol.). After about an hour's further stirring at room temperature, neutralization is carried out with 30 per cent. aqueous potassium carbonate solution. The ether solution is dried for two hours over potassium carbonate free from water and the solvent is removed under slightly sub-atmospheric pressure. After a short forerun the reaction distils over as a colourless liquid having a boiling point of 40—42°/32mm. The yield of methyl-*N*-*isobutyl*-*isonitrone* amounts to 47 gm. (41% of theory). The compound has the formula

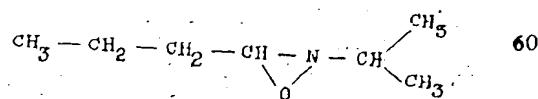


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$\text{C}_6\text{H}_{13}\text{ON}$  (115.1); Calculated, C: 62.60; H: 11.38; N: 12.16; Found, C: 63.67; H: 11.67; N: 11.50.

## EXAMPLE 2.

72 Grams (1 mol.) *n*-butyraldehyde are added drop by drop whilst stirring at —20° C. to —10° C. inner temperature to a solution of 59 gm. (1 mol.) isopropylamine in 200ml. of ether. After one hour there are added in a similar manner 100gm. of 76% peracetic acid (1 mol.); after 3 hours standing at room temperature neutralization is carried out with 30 per cent. potassium carbonate solution. Drying is effected over potassium carbonate free from water, the ether distilled at normal pressure and the *isonitrone* thus formed is distilled in a water jet vacuum. There are obtained 92gm. of a colourless liquid having a boiling point of 43—45° C./13mm. The yield of *n*-propyl-*N*-*isopropyl* *isonitrone* amounts to 71% of theory. The compound has the formula



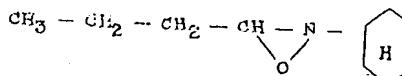
$\text{C}_6\text{H}_{11}\text{ON}$  (129.2); Calculated, C: 65.07; H: 11.70; N: 10.84; Found, C: 65.30; H: 11.89; N: 10.35.

The corresponding *isonitrone* from equimolar proportions of *n*-butyraldehyde and *N*-*isopropyl* hydroxylamine boils at 87—89° C./9mm.

## EXAMPLE 3.

To a solution of 198gm. (2 mols.) cyclohexylamine in 400ml. ether there are added drop by drop with stirring at an inner temperature lying between —20 and —10° C., 144gm. (2 mols.) *n*-butyraldehyde. After one hour there are added similarly 207 gm. 73.4% peracetic acid (2 mols.). After five hours standing at room temperature neutralization is carried out by means of ice cold 30 per cent. potassium carbonate solution and drying is carried out over solid potassium carbonate. After removal of the solvent in vacuum, the *n*-propyl-*N*-cyclohexyl-*isonitrone* distils at 78—80° C./0.8mm. The yield amounts to 220gm. (65% of the theory).

The compound has the formula



$\text{C}_{10}\text{H}_{19}\text{ON}$  (169.3); Calculated, C: 70.96; H: 11.32; N: 8.28; Found, C: 71.15; H: 11.45; N: 8.44.

The corresponding *isonitrone* obtained from *n*-butyraldehyde and *N*-cyclohexyl-hydroxylamine boils at 103—104° C./0.02mm.

34 Grams of the *isonitrone* is warmed in a 2 litre flash with a wide tall mounting duct until the violent conversion reaction starts. The dark reaction product is then distilled by fractionation. At 90—95° C./0.8mm. there are obtained 14.5gm. of a liquid fraction and at 135—139° C./0.7mm. there are obtained 12.5gm. of a solidifying fraction. The crystalline fraction is identical with the reaction product obtained from butyrylchloride and cyclohexylamine and thus consists of *N*-cyclohexyl-*n*-butyramide having a melting point of 62—63° C. The liquid fraction which is isomeric with the crystalline fraction consists of *N*-formyl-*N*-*n*-propyl-cyclohexylamine. By saponification with 20 per cent. hydrochloric acid there is obtained therefrom a base similar to that obtained by hydrogenating *N*-*n*-propyl-aniline.

## EXAMPLE 4.

To a solution of 148gm. (2.5 mol.) *iso* propylamine in 1 litre of anhydrous acetone, to which is added 250gm. anhydrous sodium carbonate. Here are added with stirring at

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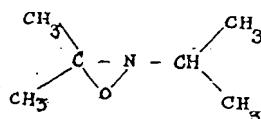
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5      -20 to 0° C. inner temperature 225gm. 84% peracetic acid (2.5 mols.). After a further 20 hours stirring at room temperature the sodium carbonate formed is removed with suction and washed out with acetone. The acetone solution is dried over potassium carbonate, the excess of acetone is removed in vacuum and the isonitrone, a colourless liquid, distilled at 74-76° C./0.4 mm. The yield of 10 dimethyl-N-isopropyl-isonitrone amounts to 170gm. (59% of theory).

10      The compound has the formula

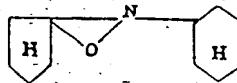


15      C6H13ON (115.2); Calculated, C: 62.57; H: 11.38; N: 12.16; Found, C: 62.31; H: 11.41; N: 12.09.

EXAMPLE 5.

20      165 Grams (1 mol.) cyclopentylidene-cyclohexylamine (boiling point 92° C./3mm.) in 400ml. of ether are reacted with 90 gm. 85% peracetic acid (1 mol.) with stirring at 0° C. After 24 hours standing at room temperature the solution is neutralised with 30% potassium carbonate and dried over potassium carbonate. 25 The solvent is removed and the isonitrone is distilled at 74-76° C./0.4mm. The yield of tetramethylene - N - cyclohexyl - isonitrone amounts to 157gm. (87% of theory).

25      The compound has the formula

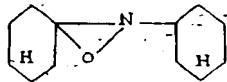


30      C11H19ON (181.3); Calculated, C: 72.87; H: 10.56; N: 7.72; Found, C: 72.63; H: 10.54; N: 7.71.

EXAMPLE 6.

35      179 Grams (1 mol.) cyclohexylidene-cyclohexylamine (boiling point 82-84° C./0.6mm.) in 400ml. of ether are reacted with stirring at 5° C. inner temperature with 90gm. 85% peracetic acid (1 mol.). After standing at room temperature for five hours neutralization with 30% potassium carbonate solution is carried out, and drying is effected over sodium sulphate. After distilling off the solvent the 40 pentamethylene - N - cyclohexyl - isonitrone, a colourless liquid, distils at 102-104° C./1.5mm. The yield amounts to 166 gm. (85% of theory).

45      The compound has the formula



50      C12H21ON (195.3); Calculated, C: 73.80; H: 10.84; N: 7.17; Found, C: 74.03; H: 10.76; N: 7.17.

55      A solution of 98 gm. of the isonitrone in 150ml. tetraline is heated in a 2 litre flask to which has been connected a mounting duct until the violent conversion reaction starts. After the reaction has subsided the reaction product is distilled at 130-132/0.4mm. There is obtained N-cyclohexyl-caprolactam. Melting point 52-53° C. The yield amounts to 83gm. (85% of theory).

60      C12H21ON (195.3); Calculated, C: 73.80; H: 10.84; N: 7.17; Found, C: 73.81; H: 10.78; N: 7.26.

EXAMPLE 7.

65      To a mixture of 98gm. (1 mol.) cyclohexanone, 159gm. (1 mol.) cyclohexylamino-acetate and 250ml. of ether there are added with stirring at 0-10° C. inner temperature 98gm. 85% peracetic acid (1 mol.). After 24 hours standing at room temperature neutralization is carried out by washing with 35% potassium carbonate solution. Drying carried out over potassium carbonate is followed by distillation, there are obtained at 90-92° C./0.3mm. 164gm. of pentamethylene-N-cyclohexyl-isonitrone (84% of theory).

70      EXAMPLE 8.

75      To a mixture of 99gm. (1 mol.) cyclohexylamine and 100gm. sodium carbonate free from water are added 98gm. (1 mol.) cyclohexanone whereby the temperature rises to 30-40° C. After cooling to room temperature dilution is carried out with 250ml. of ether and the product is reacted with stirring at 0° C. inner temperature with 106gm. 72% peracetic acid (1 mol.). After 24 hours stirring at room temperature the salts are dissolved by the addition of water, and the ether solution is dried over potassium carbonate. The ether is removed and the pentamethylene-N-cyclohexyl-isonitrone is distilled at 95-98° /0.6mm. The yield amounts to 174gm. (89% of theory).

80      EXAMPLE 9.

85      To a mixture of 49gm. (½ mol.) cyclohexanone, 49.5gm. (½ mol.) cyclohexylamine and 1gm. magnesium hydroxide there are added drop by drop, with stirring, 57gm of 30% hydrogen peroxide (½ mol.) whereby the temperature rises to 100° C. After a further three hours heating to boiling point, the cooled reaction mixture is extracted with ether. After distillation of the ether solution dried over sodium sulphate, there are obtained at 100-104° C./1.2mm. 30 gm. of pentamethylene-N-cyclohexyl-isonitrone. The yield amounts to 31% of theory.

90      EXAMPLE 10.

95      153 Grams (1 mol.) cyclohexylidene-isobutylamine (boiling point 92-94° C./20mm.) in 400ml. of ether are reacted at 0° C. inner temperature with stirring with 96gm. of 85% peracetic acid (1 mol.). After 10 hours standing at room temperature neutralization is carried out with 30% potassium carbonate and drying is effected over potassium carbo-

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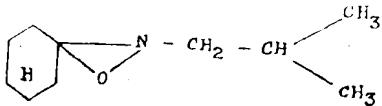
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5 nate. After removal of the ether the penta-methylene-N-isobutyl-*isonitrone* distils at 55-57° C./0.6mm. The yield amounts to 150gm. (89% of theory).

5 The compound has the formula

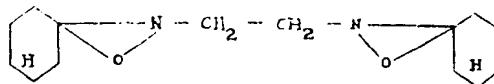


10  $C_{10}H_{19}ON$  (169.3); Calculated, C: 70.95; H: 11.31; N: 8.27; Found, C: 71.35; H: 11.43; N: 8.28.

EXAMPLE 11.

10 To a solution of 60gm. (1 mol.) ethylenediamine in 600ml. of ether to which has been added 140gm. anhydrous potassium carbonate, there are added drop by drop with stirring at 0-5° C. inner temperature 196gm. (2 mol.) cyclohexanone. After two hours standing there are added with stirring at -10 to 0° C. inner temperature, drop by drop, 200gm. 76% peracetic acid (2 mol.). After 30 hours stirring at room temperature cooling is effected to -20° C. The crystalline mass which has precipitated is suction filtered off and washed with water. After drying there are obtained 96gm. of the di-pentamethylene-N-N'-ethylene-di-*isonitrone* having a melting point of 105-106° C. which after recrystallisation from ether or benzine containing light fractions melting at 106-107° C. The yield amount to 39 per cent. of theory.

20 The compound has the formula



25  $C_{14}H_{22}O_2N_2$  (252.3); Calculated, C: 66.63; H: 9.59; N: 11.10; Found, C: 66.39; H: 9.65; N: 11.10.

EXAMPLE 12.

30 80 Grams ( $\frac{1}{2}$  mol.) cyclopentylidene-aniline (boiling point 86-90° C./0.4mm.) in 250ml. of ether are reacted drop by drop at -20° C. to -10° C. with 50gm. 76% peracetic acid ( $\frac{1}{2}$  mol.) with stirring. After one hour a dark brown solution which contains the tetra-methylene-N-phenyl-*isonitrone* is shaken with ice cold potassium carbonate until neutral, dried for one hour in anhydrous potassium carbonate and freed from the solvent in vacuum. The ether residue is heated in a 2 litre flask to which a mounting duct has been connected until the exothermic conversion reaction takes place. By distillation of the dark coloured reaction product there are obtained at 135-138° C./0.6mm., 27gm. N-phenyl-piperidine-2, having a melting point of 99-100° C. The yield is 31% of theory.

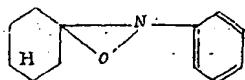
35 The constitution of the compound is confirmed by determination of the melting point

of a mixture with N-phenyl-piperidine-2 obtained by partial electrolytic reduction of N-phenyl-glutarimide according to Buhei Sakurai (Ref. Chem. Abstr. 32, 8281 (1938)).

EXAMPLE 13.

40 58 Grams ( $\frac{1}{3}$  mol.) cyclohexylidene-aniline (boiling point 150-152° C./21mm.) in 200ml. of ether to which have been added 50gm. anhydrous potassium carbonate are reacted with stirring at 0° C. inner temperature with 26.7gm. 95% peracetic acid ( $\frac{1}{3}$  mol.). After one hour stirring at room temperature the salts are brought into solution by the addition of water. The ether solution is dried with potassium carbonate and the ether is removed in vacuum. There is obtained in practically quantitative proportion the penta-methylene-N-phenyl-*isonitrone* which melts after recrystallisation from benzine containing light fractions at 75° C. The product has the odour characteristic of quinone and is explosive.

45 The compound has the formula



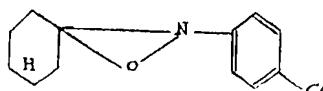
50  $C_{12}H_{15}ON$  (189.3); Calculated, C: 76.15; H: 7.99; N: 7.40; Found, C: 76.33; H: 8.17; N: 7.57.

55 6.5 Grams of the *isonitrone* are heated to the boiling point in 60ml. of xylene in a flask, to which a condenser has been connected until the conversion reaction starts. After distilling off the solvent the N-phenyl-caprolactam which has been formed remains as a crystalline mass. By distillation at 150° C./1.2mm. are obtained 5gm. of the pure N-phenyl-caprolactam. Melting point 75° C.

60  $C_{12}H_{15}ON$  (189.3); Calculated, C: 76.15; H: 7.99; N: 7.40; Found, C: 76.17; H: 8.15; N: 7.36.

EXAMPLE 14.

65 21 Grams of cyclohexylidene-*p*-chloro-aniline (boiling point 108-110° C./0.2mm.) in 150ml. of ether are reacted at -20° to -15° C. with stirring with 10gm. 76% peracetic acid. After one hour standing at 0° C. the brown solution is washed with cold potassium carbonate solution and dried over potassium carbonate. After removal of the solvent in vacuum there are obtained 70gm. of crystallised pentamethylene-N-(*p*-chlorophenyl)-*isonitrone* which may be purified by recrystallisation from ether or from benzine containing light fractions with addition of animal charcoal. Melting point 69-70° C. The compound has the formula



$C_{12}H_{14}ONCl$  (223.7); Calculated, C: 64.43; H: 6.31; N: 6.26; Found, C: 64.69; H: 6.40; N: 6.86.

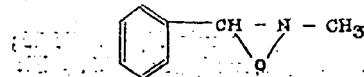
By heating 10gm. of the isonitrone in 50ml. of xylene with reflux until the conversion reaction starts there are obtained, after distillation, 9gm. of N-(*p*-chlorophenyl)-caprolactam (boiling point 170–172° C./0.8mm.). Melting point 68–69° C.

$C_{12}H_{14}ONCl$  (223.7); Calculated, C: 64.43; H: 6.31; N: 6.26; Found, C: 65.20; H: 6.67; N: 6.99.

EXAMPLE 15.

A solution of 119gm. (1 mol.) benzylidene-methylamine (boiling point 72–73° C./13mm.) in 500ml. benzene is reacted with stirring at an inner temperature of 15 to 20° C. with 140gm. 74 per cent. perbutyric acid (1 mol.). After 20 hours standing at room temperature neutralization is carried out with 30 per cent. potassium carbonate solution. The benzene layer is dried over potassium carbonate and the solvent is removed in vacuum. The phenyl-N-methyl-isonitrone distils at 70° C./2.5mm. The yield amounts to 88gm. (65% of theory).

The compound has the formula



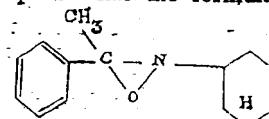
$C_8H_9ON$  (135.2); Calculated, C: 71.09; H: 6.71; N: 10.36; Found, C: 71.36; H: 6.72; N: 9.85.

The corresponding nitrone obtained from benzaldehyde and N-methylhydroxylamine melts at 84–86° C. and boils at 125° C./1mm.

EXAMPLE 16.

100 Grams ( $\frac{1}{2}$  mol.)  $\alpha$ -methyl-benzylidene-cyclohexylamine (boiling point 112–114° C./0.6mm.) in 500ml. of ether are reacted at –10° C. to 0° C. inner temperature with 45gm. 85% peracetic acid ( $\frac{1}{2}$  mol.). After two hours standing at room temperature washing is carried out with 30% potassium carbonate solution and drying is effected over potassium carbonate. By distillation of the reaction product freed from the solvent there are obtained, at 118–120° C./.8mm., 66gm. of methyl-phenyl-N-cyclohexyl-isonitrone. The yield amounts to 61% of theory.

The compound has the formula

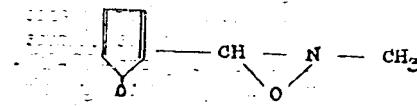


$C_{14}H_{14}ON$  (217.3); Calculated, C: 77.38; H: 8.81; N: 6.45; Found, C: 77.91; H: 8.99; N: 6.44.

EXAMPLE 17.

109 Grams. (1 mol.) furfurylidene-methylamine (boiling point 58° C./17mm.) in 500ml. of ether are reacted at 0° C. inner temperature with 88gm. 86% peracetic acid (1 mol.). After 2 hours standing whereby the solution reaches room temperature neutralisation is effected with 30 per cent. potassium carbonate solution and the ether solution is dried for 2 hours over potassium carbonate. The  $\alpha$ -furyl-N-methyl-isonitrone, a colourless liquid, is freed from the solvent in vacuum and is distilled at 44–46° C./0.6mm. The yield amounts to 66gm. (53% of theory).

The compound has the formula



What we claim is:—

1. Process for producing isonitrone as herein defined which comprises reacting an aldimine or ketimine with a per-compound.

2. Process for producing isonitrone as hereinbefore defined which comprises reacting a mixture of an aldehyde or ketone and a primary amine or a salt thereof, with a per-compound.

3. Process according to claim 1 or 2 in which the per-compound is a per-acid.

4. As new compounds, isonitrone as herein defined.

5. N-substituted carboxylic acid amides when obtained by re-arrangement of the isonitrone claimed in claim 4.

6. Process for producing isonitrone substantially as described in any of the foregoing Examples.

CARPMAELS & RANSFORD,  
Agents for Applicants,  
24, Southampton Buildings, London, W.C.2.

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